



August 15, 2014

File No.: 01-773180-000

Dr. Laura C. Buelow
Project Manager, Hanford/INL Project Office
U.S. Environmental Protection Agency, Region 10
309 Bradley Boulevard, Suite 115
Richland, WA 99352

Mr. Matt Wilkening
Project Manager, Idaho Office
U.S. Environmental Protection Agency, Region 10
950 W. Bannock Street, Suite 900
Boise, ID 83702

VIA ELECTRONIC MAIL ONLY

Subject: Upper Columbia River Remedial Investigation Feasibility Study - Phase 2
Sediment Study; Backscatter Electron Microscopy Technical Memorandum

Dear Dr. Buelow and Mr. Wilkening:

Consistent with the U.S. Environmental Protection Agency (EPA) approved Quality Assurance Project Plan (QAPP) for the above-referenced study; Teck American Incorporated (TAI) is pleased to submit for your review and approval the following technical memorandum outlining the proposed approach to conduct backscatter electron microscopy (BSEM) on archived sediments. The approach herein addresses Section A-9 of the QAPP in that samples be selected for this specialized work following a review of the preliminary chemistry data; and be documented in a technical memorandum, or QAPP addendum, for EPA's review and approval.

This technical memorandum summarizes the proposed samples selected for analysis; the methodology; and the selected laboratory including qualifications.

Sample Selection

Samples were selected on the basis of a range of predicted slag content as determined by metal ratios in accordance to Section A7.2 of the QAPP: “Can the nature and extent of unacceptable risk at the Site via spatial gradients and sediment bed properties such as slag content (e.g., Zn/V ratio¹), TOC, mPECQ, and sediment texture be further refined²?” As noted on page A-9 of the QAPP, “Sediment samples will be archived and no fewer than 35 sediment samples will undergo backscatter electron microscopy following a review of the preliminary data.” Accordingly, TAI proposed 38 samples for BSEM analysis in a letter to EPA dated February 25, 2014 (Attachment A). EPA responded via email on June 4, 2014 with a memorandum requesting an additional four samples be added to the 38 identified by TAI (Attachment A). Rationale for selection of these samples is included in Attachment A. ALS Environmental (ALS) has confirmed that sample matrix exists for all 42 samples. A list of samples to be analyzed for BSEM is presented as Table 1.

Method Overview

TAI will evaluate elemental composition of the identified samples using scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS). The SEM analytical device, in backscattered imaging mode, directs a beam of electrons at a prepared sample. Between 20-30% of the beam electrons collide with atoms comprising the sample, and rebound elastically (with little energy loss) backscattering out of the specimen. These re-emergent beam electrons are known as backscattered electrons. The production of backscattered electrons is a function of the atomic number of the atoms comprising the sample specimen. Because of this relationship, phases differing by average atomic number can be distinguished and consequently imaged relative to the phase of the backscattered electron; e.g. brightness is proportional to the average atomic number. This image will be processed by computer (computer controlled scanning electron microscopy - CCSEM) and compiled into an electronic file summarizing each sample particle's elemental composition. From this data, particle phases can be inventoried and reported.

Specific Method

- A minimum of 200 grams of sample matrix archived at ALS will be transferred under chain of custody to the BSEM laboratory.

¹ The basis and rationale of using a Zn/V ratio was detailed within Appendix D of the BERA work plan (TAI 2011). Other chemical ratios and/or methods (i.e., backscatter electron microscopy) may also be used to refine sediment bed properties and facilitate data interpretation.

² The sampling design is not intended to provide an assessment of spatial distribution of contaminants in the Site.

- Upon receipt, the BSEM laboratory will dry each sample in a low temperature oven.
- Samples will be weighed, and then sieved using a 2-millimeter screen. The coarse fraction will be visually observed for presence of black particles (e.g., slag), weighed, and retained. The finer fraction passing the 2-millimeter sieve will be weighed and retained for BSEM analysis.
- The retained sample will be split using rotary-split or other appropriate method. A sub-sample of one split will be collected and prepared into an epoxy grain mount and polished. The remainder will be archived.
- Based on Zn:V ratios, the BSEM laboratory will conduct initial analyses using low (LAL-5; Zn:V = 2.33) and high (SE-3-R8; Zn:V = 493.7) Zn:V ratio samples to identify optimal field-of-view, image enhancement, post-imaging processing, analysis stopping criterion (typically grain count), and other calibrations and thresholds.
- Once calibrations and thresholds are optimized, the prepared sample is inspected using the SEM in the backscattered electron imaging mode, where brightness is proportional to the average atomic number. In this mode, the particles are brighter than the epoxy and are detected by the CCSEM program.
- Starting with a field of view and with a suitable magnification to include many particles, a particle is detected, its periphery defined, and the size (various diameters, perimeter and area) measured. A microimage of the individual particle is acquired using SEM; and the elemental composition is acquired using energy dispersive spectroscopy (EDS). Electronically (and automatically) each particle is classified into a specific phase (mineral, slag, etc.). Analysis continues for all particles in the field, and additional fields are analyzed until the specified stopping criteria are met.

BSEM Reporting

Based on a random grain orientation in the plane of the polished section, the area assigned to each particle phase is proportional to the volume of that phase in the bulk sample, even though the plane of the section may not pass through a particle center. The BSEM laboratory will report, by sample, total area (and, by extension, volume) proportions of the sample by particle phase, the estimated mass proportions of the sample by particle phase, and the apparent size distributions by particle phase. Gray scale (256 shades) SEM images also will be reported. These data will be incorporated into the project database and linked to results for that sample.

Quality Assurance/Quality Control

Duplicate epoxy grain mounts will be prepared for approximately 10% of the samples, which will be analyzed and reported separately. One laboratory-grade clean silica sand sample blank will be processed and analyzed for every 20 samples.

Laboratory

RJ LeeGroup, Inc., (RJLG) headquartered at 350 Hochberg Road, Monroeville, Pennsylvania, will conduct the BSEM analysis. RJLG is a private, nationally recognized, scanning electron microscopy (SEM) service provider.

RJLG's company overview is summarized in the following six slides from a PowerPoint Presentation. Attachment B presents additional RJLG corporate information:

RJLG RJ LeeGroup, Inc.
Delivering Scientific Resolution

History

- Formed from the Applied Research Arm of U.S. Steel in 1985
- Spun-Off the Following as Independent Corporations
 - RJ Lee Instruments in 1995
 - RJ Lee Education in 1998
 - RJ Lee Solutions in 2000
 - Material Service Life Partnership Established in 2000
 - RJ Lee MicroSystems in 2000
 - Delta Energy in 2005
- Grew into a \$35 million company with over 250 employees in five locations by 2007
- Added 150 Employees with the Hanford Support Facility in 2009
- Exemplar projects
 - Characterization of asbestos and in situ asbestos reduction
 - Computer controlled scanning electron microscope
 - Concrete infrastructure lifespan modeling
 - Delta Energy tire program
 - World Trade Center clean up and other Post-Disaster Assessments
 - Federated architecture for Air Force
 - Mission Support Contract at Hanford Nuclear site

RJLG RJ LeeGroup, Inc.
Delivering Scientific Resolution

RJLG: Our People

RJLG employs over 300 professionals with backgrounds in various disciplines:

- Biology
- Chemical Engineering
- Chemistry
- Civil Engineering
- Computer Science
- Electrical Engineering
- Environmental Sciences
- Geology
- Industrial Hygiene
- Materials Science
- Mathematics
- Mechanical Engineering
- Metallurgy
- Mineral Processing
- Mineralogy
- Physics

Over 1,500 technical papers published and presented
Memberships in more than 30 professional societies

RJLG RJ LeeGroup, Inc.
Delivering Scientific Resolution

Core Competencies

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graph TD
    PE[Professional Expertise] --- CIT[Scientific Innovation & Technology]
    AS[Analytical Services] --- CIT
    IDM[Information & Database Management] --- CIT
    CIT --- CIT
  
```

RJLG RJ LeeGroup, Inc.
Delivering Scientific Resolution

Service Areas





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graph TD
    MC[Materials Characterization] --- SIT[Scientific Innovation & Technology]
    NT[Nanotechnology] --- SIT
    GO[Government] --- SIT
    SI[Sustainable Infrastructure] --- SIT
    HS[Health Services] --- SIT
    EN[Energy] --- SIT
    SIT --- SIT
  
```

RJLG RJ LeeGroup, Inc.
Delivering Scientific Resolution

Instrumentation

RJLG is a full service analytical lab with many in-house capabilities:



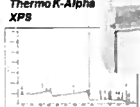
 SEM/CCSEM
 ICP and CVAA
 TEM
 GC/MS and HRGC/HRMS



- Auger & ESCA
- Atomic Absorption
- Gravimetric
- HPLC
- ICP and ICP/MS
- Infra-red Spectroscopy
- Microscopy
- Metallography
- DSC / TGA
- OC/EC (Diesel)
- UV-Vis Spectroscopy
- X-ray Diffraction
- X-ray Fluorescence

RJLG RJ LeeGroup, Inc.
Delivering Scientific Resolution

Advanced Instrumentation

Cutting Edge Technology
RJ Lee Group utilizes innovative technology for unprecedented materials characterization abilities.

 Hitachi HD-2300 STEM
 Hitachi S-5500 SEM/STEM
 Thermo K-Alpha XPS

 Hitachi HD-2300 STEM
 Thermo K-Alpha XPS

RJLG's Experience in Particle Analysis

Over its almost 30 years of operation, RJLG has had a significant portion of its work in the characterization of individual particles. Examples include:

- SEM evaluation of outdoor and indoor airborne particulate and dust, building materials (including asbestos), soil, and inclusions in metals. Representative project descriptions are presented in Attachment B.
- Beginning in 1991 under the direction of Dr. John Drexler and Region 8 EPA, RJLG characterized heavy metals (mostly lead) in soils in and around Leadville, Colorado. As part of this project hundreds of samples were analyzed. The project was managed by Dr. Stephen Kennedy, the proposed RJLG project manager for the UCR sediment BSEM analysis. Dr. Kennedy's *curriculum vita* is presented in Attachment B.
- Several published papers authored by Dr. Kennedy are included in Attachment B to demonstrate RJLG's and Dr. Kennedy's ability to conduct BSEM analysis.

RJLG's Instrumentation Relative to Project

- Sample preparation equipment includes particle mount (various), polished section, and thin section preparation tools.
- RJLG's scanning electron microscopy units, all with Secondary Electron Imaging (SEI) and Backscattered Electron Imaging (BEI) capabilities:

Instrument	Capabilities	Number of Units
ASPEX PSEM 75 ^{1,2}	SEM-EDS, 3 VP	10
JEOL 6460	VP SEM-EDS	1
Tescan Vega ¹	SEM-EDS	2
Tescan Mira TC ¹	FESEM-EDS	1
Hitachi S5500	FESEM/STEM-EDS	1
Hitachi HD2300A	FESTEM-EDS	1

¹ These instruments are capable of CCSEM

² RJLG designed and manufactured the original PSEM 75 later acquired by ASPEX Instruments.

We would like to thank you in advance for your attention to this matter and look forward to receiving your approval in the very near future. Should you have any questions or require any additional information at this time, please call me at (509) 623-4501.

Sincerely,

Teck American Incorporated



Kris R. McCaig

Manager, Environment and Public Affairs

cc: Dr. Nicholas Gard – Exponent, Inc.; Bellevue, WA
Dave Enos – Teck American Incorporated, Spokane, WA

Table 1. Summary of Sediment Samples for Specialized Backscatter Electron Microscopy Analysis and Estimated Archived Mass

Sample Id	River Mile	Estimated Archived Mass (g)
LAL-5	Canada	2800
SE-1-R5	744	1575
SE-1-R1	744	1470
SE-1-R2	742	1470
SE-1-B5	737	1470
SE-1-R8	737	1470
SE-1B-R3	735	1470
SE-1B-C3	735	1470
SE-1B-C1	734	2870
SE-2-B1	733	1470
SE-2-B2	732	1470
SE-2-R1	732	2940
SE-2-R3	732	1470
SE-2B-R1	728	1470
SE-2B-C4	728	1470
SE-2B-C3	726	1470
SE-3-B1	725	893
SE-3-C1	724	1470
SE-3-B2	724	280
SE-3-B4	723	333
SE-3-R7	722	1470
SE-3-R8	722	1470
SE-3-R10	721	595
SE-3-R9	721	1470
SE-3-C4	721	1470
SE-3B-C3	715	1470
SE-4-R1	711	1470
SE-4-B1	710	1470
SE-4-B6	709	1470
SE-4-B2	708	1470
SE-4-B4	706	1470
SE-4-B5	705	1470
SE-4-C4	705	1470
SE-4B-C3	692	1470
REF-3	689	1300
SE-5-B2	678	2800
SE-5B-C1	674	1470
SE-6-B4	665	3500
SE-6B-C4	652	1470
SE-7-B1	646	1470
SE-8-B3	605	1470
SE-8B-C2	600	1470

Attachment A
BSEM Sample Selection Correspondence



February 25, 2014

File No.: 01-773180-000

Dr. Laura C. Buelow
Project Manager, Hanford/INL Project Office
U.S. Environmental Protection Agency, Region 10
309 Bradley Boulevard, Suite 115
Richland, WA 99352

Mr. Matt Wilkening
Project Manager, Idaho Office
U.S. Environmental Protection Agency, Region 10
950 W. Bannock Street, Suite 900
Boise, ID 83702

VIA ELECTRONIC MAIL ONLY

**Subject: Upper Columbia River Remedial Investigation Feasibility Study - Phase 2
Sediment Study; Backscatter Electron Microscopy Proposal**

Dear Dr. Buelow and Mr. Wilkening:

Consistent with the U.S. Environmental Protection Agency (EPA) approved Quality Assurance Project Plan (QAPP) for the above-referenced study; Teck American Incorporated (TAI) is pleased to submit for your review and approval a proposal to conduct backscatter electron microscopy on archived sediments. As noted on page A-9 of the QAPP, "Sediment samples will be archived and no fewer than 35 sediment samples will undergo backscatter electron microscopy following a review of the preliminary data."

The following proposal is intended to address the additional question outlined within Section A7.2 of the QAPP: "Can the nature and extent of unacceptable risk at the Site

via spatial gradients and sediment bed properties such as slag content (e.g., Zn/V ratio¹), TOC, mPECQ, and sediment texture be further refined²?”

To facilitate the identification of samples for consideration of backscatter electron microscopy, field observations as recorded on sampling forms (i.e., “Sediment/Porewater Sampling Form”) were reviewed to identify if and what percentage of silica glass particles were observed. Copies of all field sampling forms can be made available; but simply due to file size (i.e., ~373 MB) were not included at this time. Following a review of all field sampling forms, 38 samples were identified as having a percentage of “silica glass” as determined by a qualified person in the field. As defined within the QAPP, “a qualified person is either a Washington State Licensed Geologist (LG) or an engineer/scientist who has received site-specific training in the following: 1) identification of sedimentary deposits of the UCR basin, 2) recognition of amorphous silica-rich glass, 3) particle size and percentage estimation, 4) soil/sediment classification systems, and 5) recording of observations.”

Field observations in conjunction with preliminary sediment chemistry (e.g., zinc to vanadium ratio and mPECQ³ calculations) were used to identify 38 samples for backscatter electron microscopy, see Table 1. We wish to confirm that upon receiving EPA’s approval on the proposed samples TAI will take the necessary steps to secure a qualified contractor to perform this specialized work.

We would like to thank you in advance for your attention to this matter and look forward to receiving your approval on the proposal. Should you have any questions or require any additional information at this time, please call me at (509) 623-4501.

Sincerely,

Teck American Incorporated



Kris R. McCaig
Manager, Environment and Public Affairs

cc: Dr. Anne Fairbrother – Exponent, Inc.; Bellevue, WA

¹ The basis and rationale of using a Zn/V ratio was detailed within Appendix D of the BERA work plan (TAI 2011). Other chemical ratios and/or methods (i.e., backscatter electron microscopy) may also be used to refine sediment bed properties and facilitate data interpretation.

² The sampling design is not intended to provide an assessment of spatial distribution of contaminants in the Site.

³ mPECQ = mean Probable Effect Concentration Quotient.

Table 1. Summary of Sediment Samples Proposed for Specialized Backscatter Electron Microscopy Analysis

Sample Id	River Mile	mPECQ8	mPECQ4	Zn:V Ratio
SE-1-R5	744	3.6	6.9	357.6
SE-1-R1	744	3.0	5.6	319.5
SE-1-R2	742	5.4	10.1	382.8
SE-1-B5	737	2.3	4.6	175.4
SE-1-R8	737	2.4	4.6	233.4
SE-1B-R3	735	5.5	10.8	335.4
SE-1B-C3	735	3.2	6.4	237.1
SE-1B-C1	734	4.3	8.8	325.5
SE-2-B1	733	0.4	0.6	32.7
SE-2-B2	732	1.8	3.6	180.0
SE-2-R1	732	3.7	7.4	304.2
SE-2-R3	732	1.0	1.8	80.0
SE-2B-R1	728	3.6	7.0	274.9
SE-2B-C4	728	6.7	14.5	377.0
SE-2B-C3	726	2.7	5.0	186.5
SE-3-B1	725	5.6	10.7	381.0
SE-3-C1	724	3.6	6.9	208.0
SE-3-B2	724	9.9	19.0	479.5
SE-3-B4	723	6.4	12.4	363.9
SE-3-R7	722	3.7	6.5	212.0
SE-3-R8	722	11.7	25.6	489.9
SE-3-R10	721	6.2	11.4	333.2
SE-3-R9	721	1.3	2.3	81.3
SE-3-C4	721	5.5	10.2	296.2
SE-3B-C3	715	5.9	11.2	307.6
SE-4-R1	711	5.0	9.6	225.4
SE-4-B1	710	6.4	12.1	326.7
SE-4-B6	709	6.4	12.2	343.8
SE-4-B2	708	5.9	11.2	340.3
SE-4-B4	706	3.6	6.6	228.7
SE-4-B5	705	4.3	7.7	307.0
SE-4-C4	705	0.7	1.1	23.5
SE-4B-C3	692	0.5	0.8	11.9
SE-5B-C1	674	0.6	0.8	8.7
SE-6B-C4	652	0.4	0.5	5.4
SE-7-B1	646	0.5	0.6	6.9
SE-8-B3	605	1.1	1.5	17.3
SE-8B-C2	600	0.5	0.6	12.2
Minimum =	600	0.4	0.5	5
Average =	N/A	4	7	232
Maximum =	744	12	26	490
Count =	38	38	38	38

N/A = not applicable

McCaig Kris SPOK

From: McCaig Kris SPOK
Sent: Tuesday, March 11, 2014 4:40 PM
To: 'Buelow, Laura'
Cc: Wilkening, Matt; Cameron Irvine (cameron.irvine@ch2m.com); Anne Fairbrother - Exponent, Inc. (afairbrother@exponent.com); Nicholas Gard - Exponent (gardn@exponent.com)
Subject: RE: Backscatter samples
Attachments: 03-11-14_Table 1 Update_Backscatter Proposal_LBuelow.pdf; 03-11-14_Teck-2013-Sedtox-combined.xlsx

Laura,

Please see attached an updated Table 1 from the letter to EPA dated February 25, 2014. Thank you again for pointing out that we inadvertently entered the wrong data in the cells for mPECQ4, mPECQ8 and Zn:V ratio for the 38 samples identified in Table 1.

Also, per your request I have attached a spreadsheet containing calculated mPECQ4, mPECQ8, and Zn:V ratios, for all Phase 2 sediment samples.

Please let me know if you have any questions or need more information.

Thanks,

Kris

Kris McCaig
Manager, Environment & Public Affairs
Teck American Incorporated
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Fax: +1.509.922.8767
Mobile: +1 (b)(6)
eMail: Kris.McCaig@teck.com
www.teck.com

From: Buelow, Laura [<mailto:Buelow.Laura@epa.gov>]
Sent: Thursday, March 06, 2014 2:17 PM
To: McCaig Kris SPOK
Cc: Wilkening, Matt; Cameron Irvine (cameron.irvine@ch2m.com)
Subject: Backscatter samples

Kris,

In order to assist EPA in determining if we agree with Teck's proposed samples for backscatter electron microscopy, we would like to request some additional information.

As offered in your letter, we would like all of the field sampling forms. I realize that the file is too large to email. If you are able, a secure file transfer would seem to be the easiest way for us to get the information. Please make sure Cameron Irvine is able to have access. If you would prefer to send the forms on a CD, please send them to myself, Matt Wilkening and Cameron Irvine.

It would make our review easier if you could send us a spreadsheet containing calculated mPECQ4, mPECQ8, Zn:V, for all samples.

Also, can you clarify if data have changed since preliminary sediment data were downloaded from the TAI-Exponent database for validation? It seems that there are inconsistent mPECQ values for the same samples reported in the memo proposing samples for BSE and in the files with proposed batching samples for sediment toxicity tests.

Thank you,

Laura Buelow, Ph.D.
Project Manager
U.S. Environmental Protection Agency
Hanford Project Office
309 Bradley Blvd, Suite 115
Richland, WA 99352
Phone: 509 376-5466
Fax: 509 376-2396
E-mail: buelow.laura@epa.gov

Table 1. Summary of Sediment Samples Proposed for Specialized Backscatter Electron Microscopy Analysis

Sample Id	River Mile	mPECQ8	mPECQ4	Zn:V Ratio
SE-1-R5	744	3.5	6.7	353.9
SE-1-R1	744	3.0	5.7	325.1
SE-1-R2	742	5.5	10.6	396.8
SE-1-B5	737	1.9	3.6	161.0
SE-1-R8	737	2.1	3.9	225.2
SE-1B-R3	735	4.9	9.4	306.7
SE-1B-C3	735	2.2	4.1	185.5
SE-1B-C1	734	4.2	8.1	327.3
SE-2-B1	733	0.3	0.5	28.1
SE-2-B2	732	2.1	3.9	241.8
SE-2-R1	732	3.7	7.1	314.4
SE-2-R3	732	0.9	1.7	85.4
SE-2B-R1	728	3.5	6.7	324.3
SE-2B-C4	728	6.6	12.7	389.3
SE-2B-C3	726	2.2	4.0	177.7
SE-3-B1	725	6.2	11.9	419.9
SE-3-C1	724	3.6	7.0	202.3
SE-3-B2	724	11.7	22.7	575.5
SE-3-B4	723	6.9	13.3	388.0
SE-3-R7	722	6.6	12.8	430.1
SE-3-R8	722	10.1	19.4	493.7
SE-3-R10	721	6.8	13.1	388.6
SE-3-R9	721	1.3	2.2	89.5
SE-3-C4	721	6.5	12.8	385.3
SE-3B-C3	715	8.2	15.9	463.0
SE-4-R1	711	5.9	11.4	297.1
SE-4-B1	710	7.3	14.0	392.9
SE-4-B6	709	7.4	14.3	421.3
SE-4-B2	708	6.6	12.8	419.8
SE-4-B4	706	4.2	8.2	302.3
SE-4-B5	705	4.9	9.6	381.9
SE-4-C4	705	0.7	1.1	22.0
SE-4B-C3	692	0.6	0.8	14.2
SE-5B-C1	674	0.6	0.7	8.4
SE-6B-C4	652	0.5	0.6	5.6
SE-7-B1	646	0.5	0.6	7.0
SE-8-B3	605	1.1	1.6	17.5
SE-8B-C2	600	0.5	0.6	12.6
Minimum =	600	0.3	0.5	5.6
Average =	N/A	4	8	263
Maximum =	744	12	23	576
Count =	38	38	38	38

N/A = not applicable

McCaig Kris SPOK

From: Buelow, Laura <Buelow.Laura@epa.gov>
Sent: Wednesday, June 04, 2014 11:45 AM
To: McCaig Kris SPOK
Cc: Wilkening, Matt; Nicholas Gard (gardn@exponent.com)
Subject: BSEM Memo
Attachments: Backscatter memo to Teck FINAL..docx

Kris,

Please find attached a memo regarding the BSEM samples. Let me know if you have any questions/concerns regarding our request for 4 additional sampling in addition to the ones that Teck proposed.

I believe the next step is for you to provide EPA with a technical memo describing how the BSEM will be performed. Is that your understanding also?

Laura Buelow, Ph.D.
Project Manager
U.S. Environmental Protection Agency
Hanford Project Office
309 Bradley Blvd, Suite 115
Richland, WA 99352
Phone: 509 376-5466
Fax: 509 376-2396
E-mail: buelow.laura@epa.gov

MEMORANDUM

SUBJECT: Back-Scatter Electron Microscopy for Sediment Samples

FROM: Laura Buelow, EPA

TO: Kris McCaig, Teck American, Inc.

Summary

EPA's level of effort (LOE) for Phase 2 sediment sampling at the UCR included submitting samples for back-scatter electron microscopy (BSEM; a potential measure of slag content in sediment). Teck (TAI) proposed 38 samples for this analysis. EPA requests that 4 new samples be included in addition to those proposed by TAI. A brief discussion on the selection of these samples is shared below.

DQOs

- Data Quality Objectives (DQOs) for this analysis were to:
 - 1) Calibrate the metal ratio approach for slag characterization; and,
 - 2) As an explanatory variable for interpreting sediment tox results.

"Can the nature and extent of unacceptable risk at the Site via spatial gradients and sediment bed properties such as slag content (e.g., Zn/V), TOC, mPECQ, and sediment texture be further refined?"

The adequacy of multiple metal ratio methods for describing sediment bed properties such as slag content will be evaluated by using field observations (e.g., presence/ absence and percent of visible black silica glass particles) in conjunction with sediment chemistry. Sediment samples will be archived and no fewer than 35 samples will undergo backscatter electron microscopy following a review of the preliminary data. Samples will be selected for this specialized work following a review of the preliminary chemistry data; and will be documented in a technical memorandum, or QAPP addendum, for EPA's review and approval."

- Samples were to be selected upon the basis of a range of predicted slag content as determined by metal ratios.

TAIs Proposed Samples

- Preliminary analytical data were evaluated in consideration of TAI's proposed samples for BSEM.
- TAI proposed 38 samples for BSEM of the 137 available samples (letter from K. McCaig to L. Buelow on 2/25/14). Most samples with field observations of visual slag were selected (29 of the 32). Most (32 of 38) samples were also from the riverine reaches (i.e., upstream of Kettle Falls).
- TAI's proposed samples are generally skewed for variables assessed (e.g., TOC, mPECQ, Zn/V, and Cu/Al, river mile). This is likely due to TAI selecting samples with visual slag - which are typically associated with the riverine reaches that were generally sandy.
- Including additional samples will improve our ability to meet DQOs.
 - Samples without visually observed slag will improve our understanding of how backscatter can identify slag characteristics when slag is not visible; and/or,
 - Samples that were also submitted for bioassays provide another variable to interpret dose-responses (dependent on toxicity data - currently unavailable).

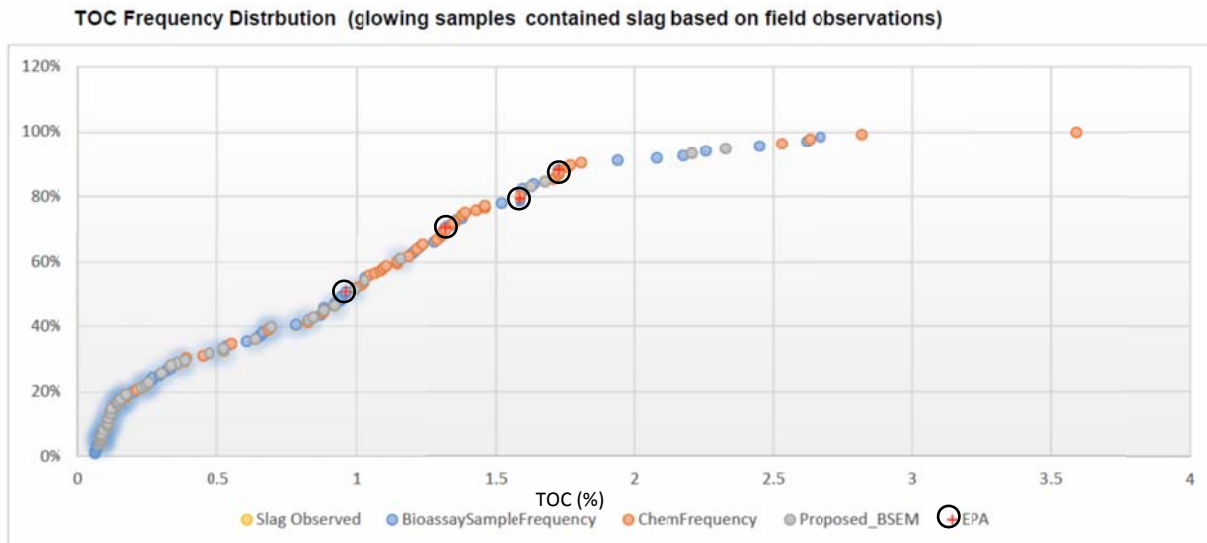
Requested Additional Analyses

EPA requests 4 additional samples be included with those proposed by TAI for BSEM analysis. LAL-5 represents a sample from an external reference site, sample 6-B4 represents a sample from Focus Area 6, and samples 5-B2 and REF-3 are samples from Focus Area 5.

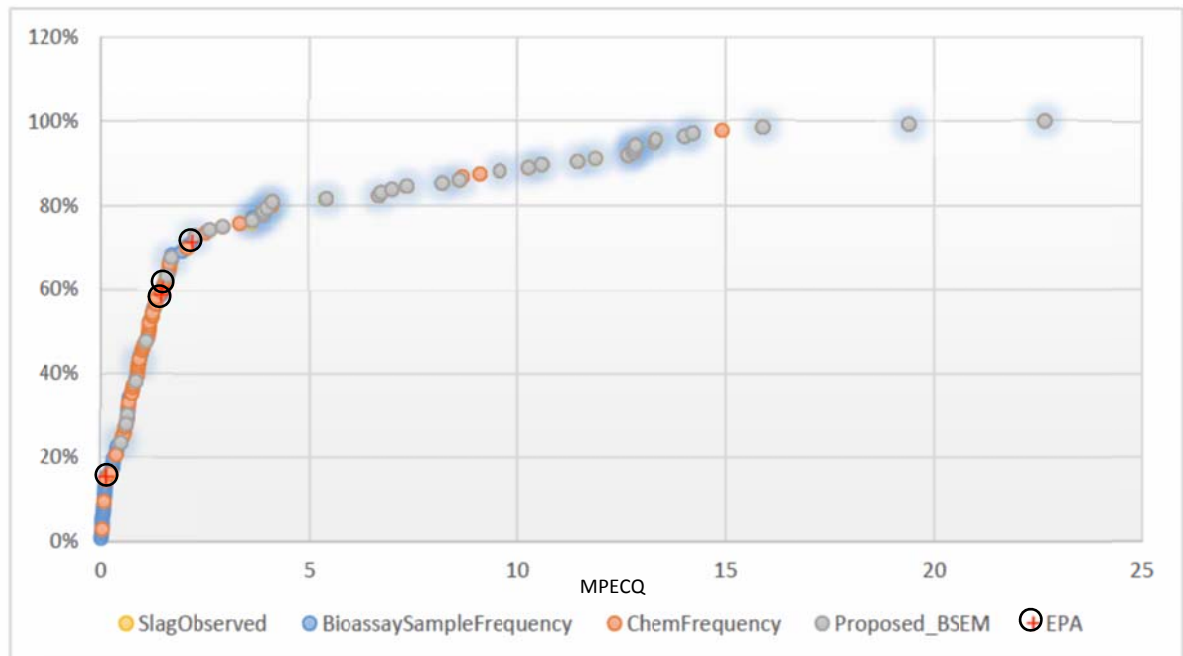
Sediment Characteristics of the 4 alternative samples for BSEM requested by EPA

Sample	RM	Visual Slag (%)	TOC (%)	mPECQ4	Silt (%)	Clay (%)	Medium Sand (%)	Zn:V	Cu:Al	Notes
6-B4	665	0	1.3	1.4	61	23	2.3	18	0.0025	From Focus Area 6.
LAL-5	Canada	0	1.7	0.14	19	19	1.3	2.3	0.00195	Canadian reference site
5-B2	678	0	1.59	2.2	57	39	1	18.7	0.0044	From Focus Area 5.
REF-3	689	0	0.961	1.5	40	12	8	19.0	0.0047	From Focus Area 5.

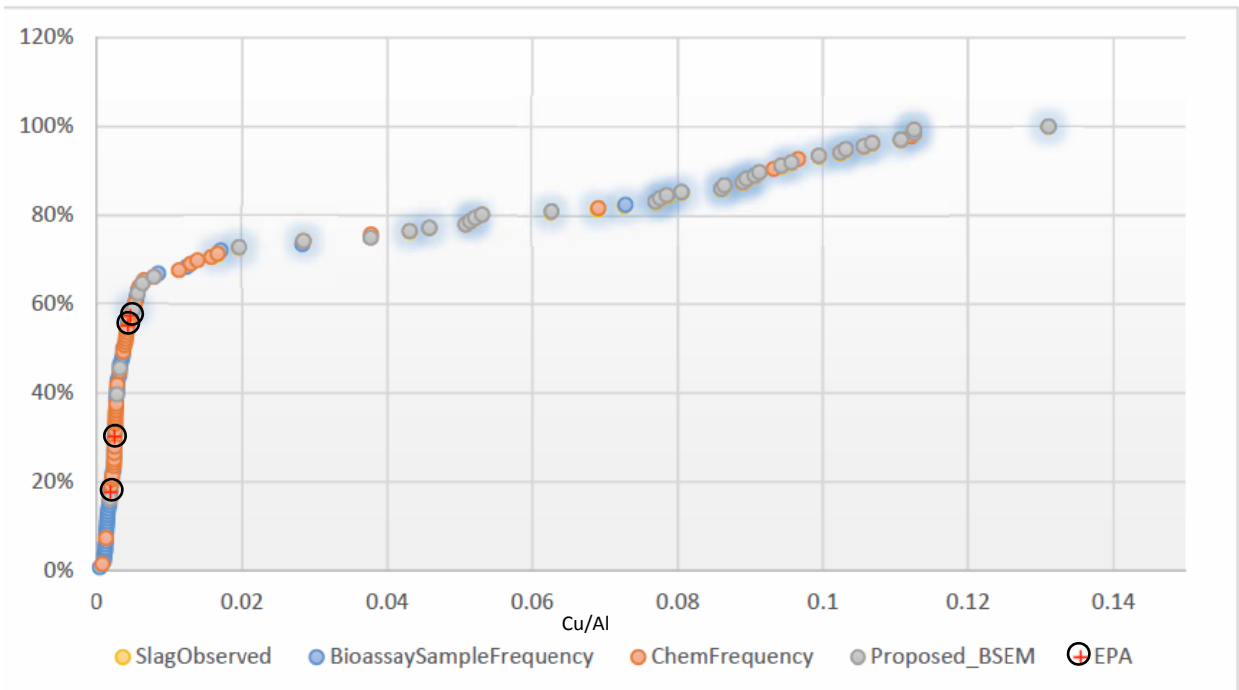
Together, these four additional samples will help meet the goal of validating visual and metal ratio methods for describing slag content as they do not have visible slag; they were sampled in reaches not described by other samples proposed for BSEM, and they cover a broader range of sediment characteristics than the TAI samples proposed for BSEM (e.g., where TOC was between 1-2.1 percent, or >2.5 percent; and, mPECQ ranged between 1-1.5 or was <0.25; see figures).



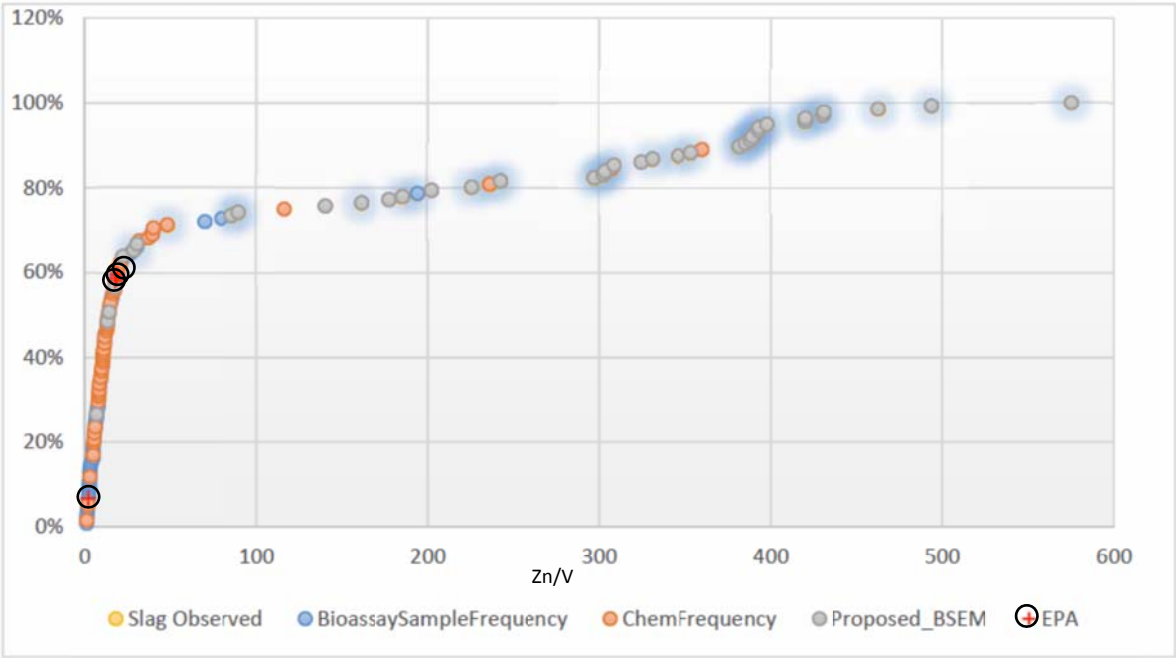
MPECQ4 Frequency Distribution (glowing samples contained slag based on field observations)



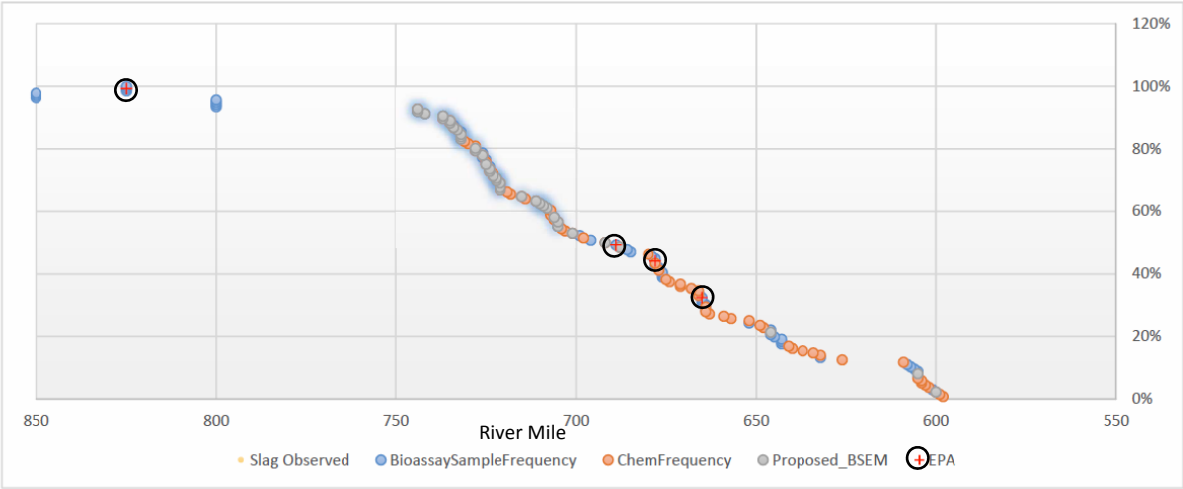
Cu/Al Frequency Distribution (glowing samples contained slag based on field observations)

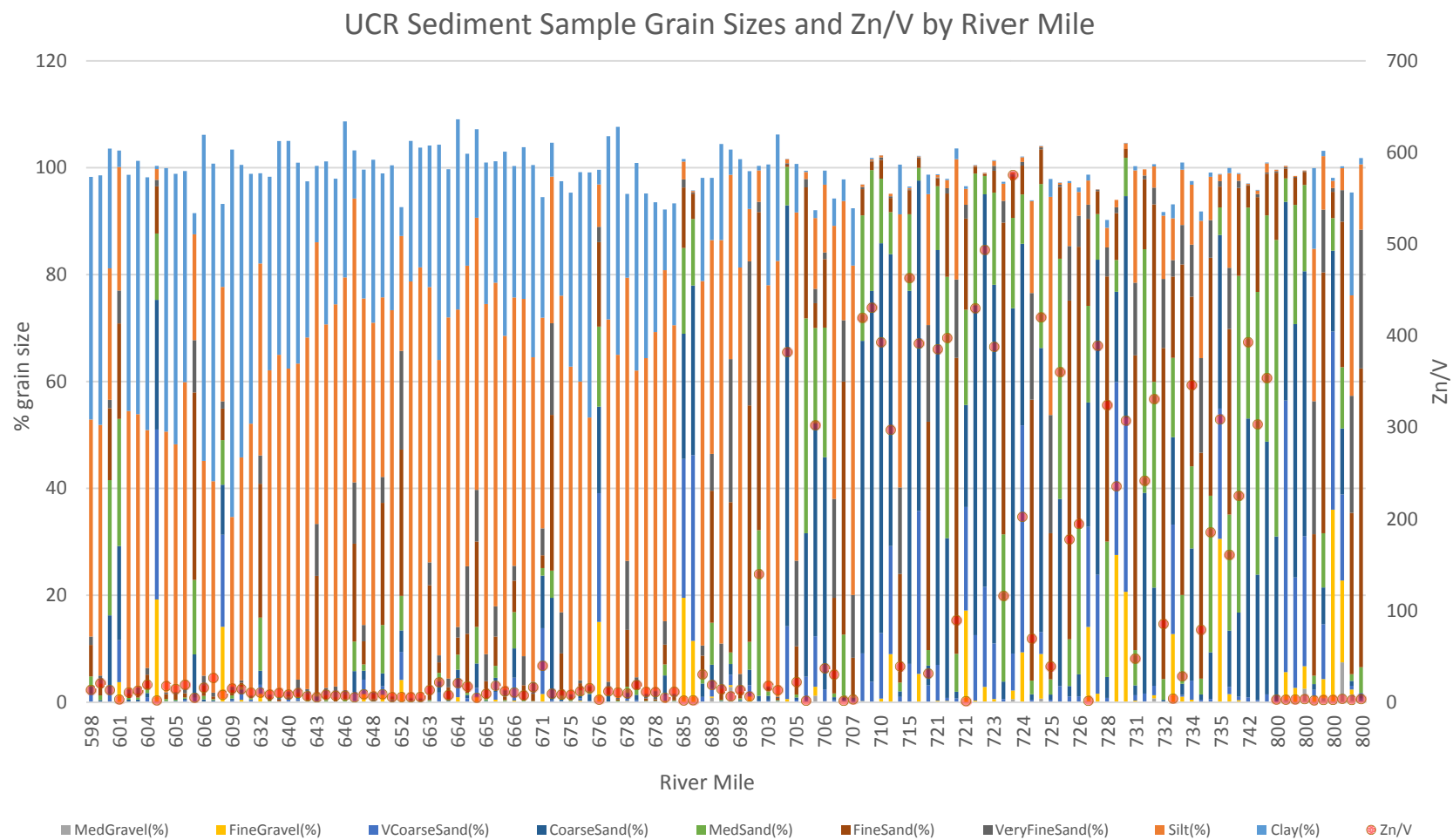


Zn/V Frequency Distribution (glowing samples contained slag based on field observations)



BSE Sampling Frequency Distribution by River Mile (glowing samples contained slag based on field observations)





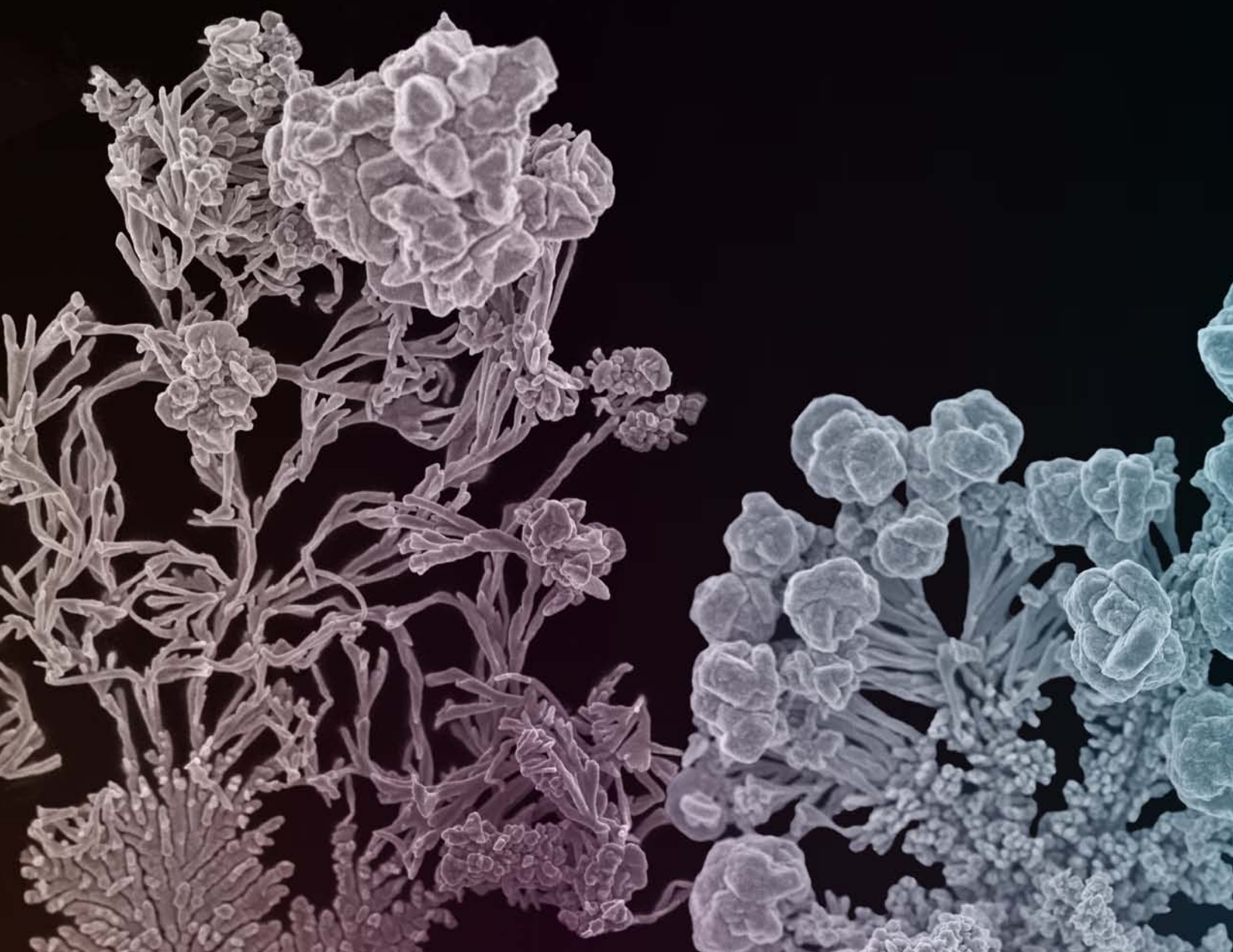
Attachment B
RJLG Qualifications



RJ LEE GROUP

DELIVERING SCIENTIFIC RESOLUTION

*We use our scientific expertise, instrumentation
and technology together with a collaborative spirit
to stimulate inquiry and solve complex problems.*



As a foremost materials characterization laboratory, RJ Lee Group delivers scientific resolution using the analytical and consultative approach of industrial forensics. We have assembled the experts and the technology platform that respond to industry at all levels of the product life cycle. We are your single source for supporting the overall continued performance of your product.

LABORATORY & TESTING SERVICES

Providing the reliable answers you need to ensure a higher measure of confidence for compliance and quality control.

INDUSTRIAL FORENSICS SERVICES

Using scientific and forensic methods to understand the problem, interpret the data and provide the solution.

LITIGATION SUPPORT SERVICES

Rendering support that is competently delivered and based on scientific authority and legal expertise.

LABORATORY INFORMATICS

Creating LIMS for laboratories and criminal forensics as well as engineering and maintenance tools for optimal software support solutions.

Our industrial forensics approach provides solutions that mitigate risk and ensure a quality product.

- Richard J. Lee, Ph.D.
CEO





INDUSTRIAL FORENSICS SERVICES

We collaborate with industry in investigative efforts to resolve materials, process, manufacturing and environmental issues.

Environmental Health & Safety

Immediate insights, protecting long-range goals, minimizing health and safety concerns that impact workers, products and the environment down to the nanoscale.

Quality Control

Achieving continuous improvement across the product life cycle. We help you gain and preserve a competitive edge in a rapidly changing regulatory landscape.

Materials Research & Development

From innovation to opportunity, we help improve upon and imagine new materials and products.

LABORATORY & TESTING SERVICES

We provide testing services to support the health and safety of workers and their environment as well as to support product and materials compliance.

Analytical Testing Services

Industrial hygiene and environmental analyses.

Onsite Sampling Services

Perform on-site, real-time analysis; support IH firms in environmental studies and post-disaster events.

Laboratory Management Services

Provide contract-based operation and management of laboratories; design customizable LIMS packages.

We understand and employ current methodologies mandated by government standards such as OSHA, NIOSH, MSHA, and the EPA.





LABORATORY INFORMATICS SOLUTIONS

Informatics solutions and software tools that simplify criminal forensics lab processes, streamline laboratory data management, and improve the reliability of engineered systems.

Themis Forensic LIMS Software

Criminal forensics software that is easy to use, collaborative in practice and adapts to your processes. Fully aligned with ISO 17025 and ASCLD standards.

Laboratory Software

Our customizable LIMS manages lab operations such as workflow and data tracking. By examining your unique requirements, we can create a package that is dependent on how you use the system.

Engineering & Maintenance Systems

Software support tools for engineers and maintenance staff that enable direct data access – a critical element in decision making for maintenance programs or as an element in determining remaining service life.

LITIGATION SUPPORT SERVICES

Robust support built on scientific data, interpreted and delivered by seasoned professionals.

RJ Lee Group maintains a visible and respected reputation within the legal community in both civil and criminal litigation support. For the last 25 years, we have assisted more than 500 law firms resolve more than 3,000 matters involving product liability, toxic tort, construction defect, nuisance dust, personal injury and patent infringement cases. Our criminal support includes hundreds of cases for gunshot residue and trace evidence including human hair comparison, paint comparison, animal hair identification and fiber identification. Our staff researches, identifies and tracks conflicts of interest, maintains proper chains of custody and organizes data productions as part of the discovery process. Immediate access to comprehensive laboratory facilities coupled with materials and environmental experts are capabilities we leverage to our clients' advantage.



OUR EXPERTISE & APPROACH

Research & Development

Provide support or collaborative effort for product development or to establish discovery through research.

Materials Characterization

Investigate the composition and structure of materials to establish material and elemental properties, internal structure, suitability for use and to explain anomalies.

Particle Characterization

Assess environmental and workplace health and safety standards; design monitoring plans, investigate, sample and identify particles; propose remediation.

Materials Selection

Recommend the materials best suited to client-specific service environments.

Nano-Characterization

Develop investigative methods and help satisfy regulatory requirements for nanomaterials. Provide QA of nanomaterials for specification conformance.

Post-Disaster Assessments

Provide sampling and analytical support to firms that assess occupational safety and health and environmental health and safety by supplementing their capabilities in the clean-up and remediation process.

Corrosion Analysis

Define a logical and systematic approach to identify corrosion mechanisms especially those that impact a product's useful life cycle.

Failure Analysis

Initiate forensic inquiry into a failed component, product or system to identify the root cause of failure and provide remedial action plan.

Heavy Metal Speciation Projects Case Studies

Case Study #1 – Elevated arsenic in residential soils.

- Background:** Residential soils near an industrial site had elevated levels of arsenic.
- Question:** Is the elevated arsenic in the residential soils due to the industrial site?
- Method of Analysis:** Computer controlled scanning electron microscopic analysis of high atomic number particles.
- Results:** Analysis of soils from the industrial site revealed arsenic to be associated with calcium, aluminum, antimony, cadmium, lead or oxygen. These occurrences were observed in a variety of sizes and morphologies. Analysis of the soils from the residential properties revealed the majority of the arsenic to be associated with oxygen, with a minor amount associated with lead. The arsenic oxide occurred as discrete particles mostly between 10 and 20 um in diameter.
- Conclusion:** The arsenic in the residential soils was not derived from the industrial site. The occurrence of relatively pure arsenic of narrow size range suggest that it was from a manufactured product such as a herbicide.

Case Study #2 – Elevated lead in residential soils.

- Background:** Residential soils near an industrial operation involving lead sulfate had elevated levels of lead in the front and side yards.
- Question:** Is the elevated lead in the residential soils due to lead and sulfur from the industrial operation?
- Method of Analysis:** Computer controlled scanning electron microscopic analysis of high atomic number particles, and manual SEM analysis of low atomic number particles.
- Results:** Analysis of high atomic number particles in soils from the residential properties revealed that the lead occurrences were mainly leaded manganese and leaded iron with minor amounts of lead phosphate and leaded barite. Only one occurrence of lead associated with sulfur was observed. Analysis of the low atomic number particles revealed bottom ash and unburned coal in addition to normal soil components.
- Conclusion:** There is no evidence to support the idea that lead in the residential soils was derived from the industrial facility. Instead it was associated with the ash of coal and was interpreted to be derived from the heating furnaces of the old residences and spread on driveways and sidewalks to enhance traction.

Case Study #3 – Source of cadmium in soils.

- Background:** Soils had elevated levels of cadmium. The soils also contained sphalerite (zinc sulfide) which is known to incorporate cadmium. Sphalerite was present in amounts far too low to be quantified by XRD.
- Question:** What proportion of the cadmium can be accounted for by sphalerite?
- Method of Analysis:** Electron microprobe analysis of the cadmium content in the sphalerite and computer controlled scanning electron microscopy to determine the amount of sphalerite in the soil.
- Results:** The electron microprobe analysis of 50 sphalerite particles from the soil revealed a average cadmium content for sphalerite in this setting to be 0.33%. The CCSEM analysis of the soil samples revealed the volume percent of sphalerite from which it's mass percent and cadmium concentrations in the sample were calculated.
- Conclusion:** On a sample by sample basis, between about 1 ppm and 26 ppm cadmium could be attributed to the sphalerite leaving the residual cadmium to be derived from other sources.

Case Study #4 – Elevated lead and arsenic in residential soils

- Background:** Residential soils near an industrial site had elevated levels of arsenic and lead.
- Question:** Identify possible sources of lead and arsenic in the vicinity of the residential neighborhood, including, but not limited to the identified industrial site.
- Method of Analysis:** Bulk chemistry, optical microscopy scanning electron microscopy
- Results:** RJLG collected samples on the perimeter of the industrial site, and from an active and an inactive railroad bed. The samples from the site perimeter were generally low in lead and arsenic. In general, samples that were high in lead and arsenic were dark gray to black and samples that contained little or no lead and arsenic were lighter brown or red. The contaminated soils were collected in close proximity to the former and active railroad line and contained ash and/or slag.
- Conclusion:** The lead and arsenic contamination of the residential soils was not due to contributions from the industrial site but was derived from slag and/or ash associated with the railroad bed.

Case Study #5 – Paint in residential soils

Background:	Residential soils near an industrial site had elevated levels of lead. Architectural paint was a suspected source as well.
Question:	What portion of the lead burden in the soil can be attributed to paint?
Method of Analysis:	Bulk chemistry, optical microscopy, manual scanning electron microscopy of paint chips and automated scanning electron microscopy analysis of the soil.
Results:	Visible paint chips picked from the soil contained layers of lead-containing paint. Lead-containing paint was also observed in the less than 2 mm fraction of the soil. Secondary (reprecipitated) lead-containing phases included phosphates and hydroxides.
Conclusion:	The majority of the lead in the residential soils can be attributed to paint. Other possible sources have been obscured by the secondary nature of the remaining lead-bearing phases.

Case Study #6 - Lead from Slag

Background:	An historic slag waste product was deployed as an exposed water barrier. Surrounding areas had elevated levels of lead.
Question:	Is the slag deteriorating and serving as a source of lead-containing sand?
Method of Analysis:	Optical microscopy and scanning electron microscopy
Results:	Samples of the slag were inspected optically to assist in identifying representative areas for SEM analysis. Polished sections were prepared and analyzed by manual SEM techniques to characterize any lead in the slag. Automated SEM analysis of the high atomic number components was performed on sand collected adjacent to the deployed slag. No lead particles or slag particles were observed in the sand.
Conclusion:	The slag did not produce observable amounts of sand size particulate upon physical and chemical weathering in the deployed environment.

General CCSEM Project Case Studies

Case Study #1 – Excessive pump wear.

- Background:** A pump manufacturer noticed a high rate of wear when pumping some fluid products compared to other products. The fluid products contained particulate.
- Question:** What is the particulate and is the wear rate related to the size or composition of the contained particulate?
- Method of Analysis:** Computer controlled scanning electron microscopic analysis was employed to characterize the particulate contained in 6 fluids.
- Results:** The total amount of particulate as well as the mass proportion and particle size for the minerals albite, quartz, nepheline and microcline were determined for each sample. These results were compared to the wear results. Surprisingly, the wear was not correlated with the largest particles or with the hardest particles, but with the total amount of particulate regardless of size and composition.
- Conclusion:** The concentration of particulate in fluid and not the size or composition of the particulate that is responsible for the wear of the mechanical pump.

Case Study #2 – Identification of metals contaminating grease.

- Background:** Grease was found to be contaminated by foreign material that compromised the effectiveness of the grease
- Question:** What is the nature of the contamination
- Method of Analysis:** Computer controlled scanning electron microscopic analysis of particles liberated from the grease.
- Results:** The particulate for three samples was liberated and characterized. Particle types included calcite, dolomite, talc, and gypsum related to building materials, as well as various metals including stainless steel, aluminum oxide, metallic copper and brass. The different samples contained different types and amounts of the contaminants.
- Conclusion:** The type and abundance of particulate were used by the client to determine the source and mechanism of contamination.

Stephen K. Kennedy

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Monroeville, PA 15146

Education:

- Ph.D., Geology, University of South Carolina, (b) (6)
- M.S., Geology, Monmouth College of Illinois, (b) (6)
- B.S., Geology, University of Illinois, (b) (6)

Career/Employment:

- RJ Lee Group, Inc., Senior Scientist, (b) (6)
- RJ Lee Group, Inc., Consulting Engineer/Scientist, (b) (6)
- University of Pittsburgh, Assistant Research Professor, (b) (6)
- University of Pittsburgh, Assistant Professor (b) (6)
- University of SC, Post Doctorate, Reservoir Evaluation via Analysis of Pore Geometry, (b) (6)
- Bureau of Land Management, Geologist, (b) (6)
- Wright College, Instructor, (b) (6)
- Lecturer, University of Illinois, (b) (6)

Summary:

- Applies manual and automated electron beam instrumentation to characterize particulate in a variety of settings, including heavy metals in soils and dust, lead and other components in paint, World Trade Center dust, etc.
- Applies optical and electron beam microscopy for the characterization of concrete components and hardened concrete.
- Major contributor to RJ Lee Group's Schoolhouse Project which specializes in bringing high-technology instrumentation into the classroom through remote access and simulation
- Particle characterization – use of morphology (quantified shape and descriptive surface texture by scanning electron microscopy), and cathodoluminescence to evaluate sediment provenance, depositional environment, and relative contributions of source terrains
- In the Department of Geology and Planetary Sciences, taught graduate courses in sandstones, mud rocks, and special topics in sedimentology. Also taught sedimentology to undergraduate geology majors and taught introductory courses in physical geology and oceanography

Certifications, Licenses, Specialized Training:

Publications & Presentations: 80

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MICROBEAM ANALYSIS OF HEAVY ELEMENT PHASES IN POLISHED SECTIONS OF PARTICULATE MATERIAL - AN IMPROVED INSIGHT INTO ORIGIN AND BIOAVAILABILITY

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Speciation and Characterization of Heavy Metal-Contaminated Soils Using Computer-Controlled Scanning Electron Microscopy

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